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Formulas (Incorporating Decomposition of Carbonates at 600 C) for the Detn. of Total Oxygen in Solid Wastes

Environmental Protection Agency

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U.S. ENVIRONMENTAL PROTECTION AGENCY

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The results obtained with these formulas are affected by the errors incurred in the data obtained by the various analyses. However, this indirect approach is satisfactory and practical with solid wastes samples.

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FORMULAS (INCORPORATING DECOMPOSITION OF CARBONATES AT 600 C) FOR THE DETERMINATION OF TOTAL OXYGEN IN SOLID WASTES

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written by
DONALD L. WILSON, Research Chemist

(Restricted distribution; submitted to
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U.S. ENVIRONMENTAL PROTECTION AGENCY
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1971

FORMULAS (INCORPORATING DECOMPOSITION OF CARBONATES AT 600 C) FOR THE DETERMINATION OF TOTAL OXYGEN IN SOLID WASTES

Donald L. Wilson
Solid Waste Management Office
Environmental Protection Agency
Cincinnati, Ohio 45213

ABSTRACT

An existing ASTM method for determining total oxygen in coal, indirectly from an ash analysis, was found not completely applicable for solid wastes samples, especially if the normal procedure of ashing solid wastes samples at 600 C is involved. With modifications of the formula in the ASTM method, total oxygen can be reasonably estimated in solid wastes samples. However, before a formula can be applied, the concentrations of total carbon, hydrogen, nitrogen, sulfur, and chlorine must be determined. Likewise, the analyst must have knowledge of the ash value for the sample and the procedure by which it was ascertained.

If the ash value is determined by muffling the sample at 600 C (normal ashing temperature for solid wastes samples) rather than at 950 C then the percent decomposition of carbonate carbon must be determined. (The ash value at 950 C may be determined with a carbon-hydrogen apparatus.)

Two modifications of the ASTM formula were required for solid wastes samples. The method used to determine the ash value dictates the appropriate formula because partial decomposition of carbonates occurs at 600 C.

The results obtained with these formulas are affected by the errors incurred in the data obtained by the various analyses. However, this indirect approach is satisfactory and practical with solid wastes samples.

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INTRODUCTION

The oxygen analysis of solid wastes is one of the important ultimate analysis necessary to determine: the efficiency of operation of an incinerator, the design of furnaces for incineration, and a complete materials balance of incoming and outgoing material. The oxygen content of solid wastes samples must be known if their calorific values are to be calculated from ultimate analyses.

The direct approach of determining oxygen content in solid wastes would involve much time and expense. Although a direct method is more exacting, an indirect approach (ASTM, 1969a) employing a formula with ultimate analyses and an ash value is presently being used for each coal sample, and this indirect approach can be applied to solid wastes samples. However, since the oxygen-formula applies to coal and coke samples and is related to a particular method of ashing (ASTM, 1969b), the formula must be modified before it can be applied to solid wastes.

The modified method is actually two oxygen formulas each of which depends upon the ashing technique employed. One procedure of ashing is the standard method for solid wastes samples (APWA, 1970) which involves heating the sample to 600 C, but, as will be shown in this paper, requires testing for the decomposition of carbonates. The other ashing technique is to weigh the ash from the carbon-hydrogen method (Wilson, 1970). Although either ashing technique can be used, ashing as part of the carbon-hydrogen method is recommended because the extension of the carbon-hydrogen procedure (weighing the residue and correcting for

oxidation of combustion aid) requires very little extra time or effort and eliminates the need for determining amount of carbonate decomposition. The effects of the two ashing procedures upon the oxygen values of solid wastes samples and upon the formulas for determining these oxygen values are discussed in this report.

APPROACH AND RESULTS

The ASTM method of determining total oxygen in coal and coke samples is by applying the following equation:

1. % oxygen (O) = 100-% carbon (C) - % hydrogen (H) - % nitrogen (N) - % sulfur (S) - % moisture - % ash

In this paper, each component of this ASTM formula will be discussed independently and some new terms will be added, thus creating the final formulas recommended for solid wastes samples.

First, the oxygen referred to in the above ASTM formula is total oxygen, including both organically combined and inorganically combined oxygen. Total oxygen content of a solid wastes sample is defined as either all the oxygen contained in the volatile-at-600 C portion plus the inorganically combined oxygen of carbonates in the sample or all the oxygen contained in the volatile-at-950 C portion. This total oxygen value does not include oxygen which is already combined with metals or silicon. In solid waste management the different types of combined oxygen may be more important than the total oxygen. For example, only organically combined oxygen is of interest in calculating the efficiency of an incinerator to dispose of organic matter.

Organically and inorganically bonded oxygen may be differentiated by performing a carbonate carbon analysis (Wilson, 1971) of the solid waste sample, and then multiply the carbonate carbon value by four to find the carbonate oxygen content. Substracting the carbonate oxygen

value from the total oxygen value (determined by one of the recommended formulas presented herein) results in the organically bonded oxygen content of the sample.

The second and third items listed in the ASTM formula are carbon and hydrogen. Although an ASTM method (ASTM, 1969c) has been commonly employed to determine total carbon and hydrogen, a carbon-hydrogen method (Wilson, 1970) explicits for solid wastes samples is recommended, particularly since this solid wastes method uses larger sample portions. Both of these carbon-hydrogen methods reveal the total carbon content of the sample and the ASTM-oxygen formula requires that the total-carbon-content value be used; however, the carbon value is related to a particular procedure for ashing the sample. Therefore, the ASTM oxygen formula is applicable with solid wastes samples only when such samples are ashed similar to coke samples, at 950 C. (The ashing temperature of coal samples, 700 to 750 C, is not recommended for solid wastes samples.) Solid wastes samples are normally volatilized at 600 C (APWA, 1970) and carbonates in such samples are partially and unpredictable decomposed at this temperature (Table I); therefore, the volatile portion of the sample does not contain all the carbon and oxygen present in the original sample.

The problem with the varying degree of decomposition of carbonates at 600 C can be overcome in two ways. One approach, the easier, is to change the normal volatile-ash method and volatilize the solid wastes samples at 950 C. At this temperature almost all carbonates present in the samples are decomposed (Tables II and III) and may be assumed completely decomposed when calculating for oxygen concentrations.

Furthermore, the data at this temperature is easily obtained by weighing the ash obtained in the carbon-hydrogen test. However, if desired, this data may be acquired by muffling the solid wastes samples (starting with a cold furnace) at 950 C for one hour in air (Table IV). Then this ash value and the total carbon value are put directly into the ASTM-oxygen formula. The 100 and % ash in the ASTM formula are now expressed as % volatiles and, for solid wastes samples, a % chlorine term is added. Thus the following formula is used for calculating total oxygen content when samples are volatilized at 950 C (data on a dry basis):

2. % total oxygen (O) = % volatiles at 950 C - % total carbon (C) - % hydrogen (H) - % nitrogen (N) - % sulfur (S) - % chlorine (Cl)

The second route in overcoming carbonate decomposition at 600 C is more difficult and complex. If the carbon-hydrogen values are already known and for some reason the ash value at 600 C is still desired, a modified formula for calculating oxygen content is needed and one more analysis is required. The carbonate carbon content of the ash from the volatilization at 600 C analysis must be determined in order to know the degree of decomposition of carbonates that has occurred. This carbonate decomposition is calculated by the following formula (data on a dry basis):

3. % decomposition of carbonates at 600 C $= \frac{(A-B)(100)}{A}$

Where: A = % carbonate carbon (Wilson, 1971) present before volatilization

B = % carbonate carbon (Wilson, 1971) Present after volatilization, corrected to original sample basis

Assuming, for calculation purposes, all carbonate oxygen is in the ash from the volatilization at 600 C, the ASTM-oxygen formula is modified in three more aspects: (a) the carbon dioxide value from decomposed carbonates is removed from the volatile value by substracting out the product of the carbon dioxide to carbon ratio and the volatilized carbonate carbon, (b) only organically bonded carbon is considered part of the volatile value, and (c) all carbonate oxygen is added to the oxygen value obtained from the volatile data. When solid wastes samples are volatilized at 600 C, total oxygen content is calculated from the following formula (data on a dry basis):

4. $\underset{(O)}{\%\ \text{total oxygen}} = \%\ \text{volatiles at 600 C} - \left(\frac{44}{12}\right)(\%\ \text{carbonate carbon}) - (\%\ \text{carbonate carbon decomposed}) - \underset{(C)}{\%\ \text{organic carbon}} - \underset{(H)}{\%\ \text{hydrogen}} - \underset{(N)}{\%\ \text{nitrogen}} - \underset{(S)}{\%\ \text{sulfur}} - \underset{(Cl)}{\%\ \text{chlorine}} + (4)(\%\ \text{carbonate carbon})$

Since the volatilization-at-600 C data for this paper was collected over a period of time, tests were conducted to determine if the volatile data varied with age of the samples. The results revealed that the volatile-at-600 C data does not change with age of sample but reproducibility is affected by amount of carbonates present (Table V).

The next item in the ASTM-oxygen formula is nitrogen. Twelve raw refuse samples were analyzed for their nitrogen content (AOAC, 1970a). The nitrogen concentration in these samples ranged from 0.49% to 1.42% and averaged 0.75% (dry basis); therefore, unless actually determined, nitrogen concentration in raw refuse samples is assumed, in this paper, to be 0.75%. The same nitrogen method was applied to nine incinerator noncombustible-residue samples. The average nitrogen content was 0.14%, ranging from 0.04% to 0.31%. Unless actually determined, the nitrogen content of noncombustible residues and similar incinerator fly ash samples is assumed to be 0.15%.

The sulfur content was determined (ASTM, 1969d) on the same 21 samples which were analyzed for their nitrogen content. The sulfur values were all about the same, ranging from 0.10% to 0.37%, and averaged 0.19%. For this paper, sulfur content of all solid incinerator samples is considered to be 0.20%, unless actually determined.

The percent chlorine content is not part of the original ASTM-oxygen formula, but is inserted for application of the formula to solid wastes samples, because often such samples have appreciable chlorine content. The average chlorine content (AOAC, 1970b) of the same 21 solid wastes samples as above varied with the type of sample involved. In raw refuse samples, the chlorine content ranged from 0.29% to 1.10% and averaged 0.52%. In the noncombustible residue samples, the chlorine content ranged from 0.06% to 0.15% and averaged 0.11%. The chlorine content, unless actually determined, is considered to be 0.50% in raw refuse samples and 0.10% in noncombustible residue (or fly ash) samples.

The ASTM-oxygen formula does include a term for moisture; however, since all solid wastes data is normally converted to a dry basis, as

in this paper, a moisture term is not included in the modified formulas.

The last term in the ASTM-oxygen formula is % ash. As previously mentioned, this item has been incorporated with the 100 into a % volatile term.

The three formulas for determining total percent oxygen have been applied to some solid wastes samples (Table VI). The greatest difference between oxygen values obtained with ASTM-oxygen formula and those obtained with the modified formulas occurs, as expected, when samples have a high carbonate content.

CONCLUSIONS

An ASTM-oxygen method was modified to determine total oxygen in solid wastes samples. The modified method consist of formulas for calculating total oxygen by correlating as many as eight different analyses. This modified oxygen method allows the ash-volatile method, which is related to the oxygen analysis, to be performed in either of the two normally used techniques, the carbon-hydrogen route at 950 C or the common practice of muffling at 600 C. The recommended procedure for determining carbonate oxygen and organically-bonded oxygen is first determine carbonate carbon, multiply this answer by four for carbonate oxygen, and then obtain the organically-bonded oxygen content by substracting the carbonate oxygen value from the total oxygen concentration.

These new formulas for oxygen concentrations still have the errors inherent in the volatile analyses, plus the other analyses involved; however, they do take into account the greatest inaccuracy, carbonate oxygen.

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The author thanks the Division of Technical Operations, Solid Wastes Office, for the provision of samples from incinerators.

TABLE I

CARBONATE DECOMPOSITION IN VOLATILE ANALYSIS
(2 hrs. @ 600 C in air)[a]

Sample No.	Type of Sample	% Carbonate Carbon		% Vol.	Corrected % Carbonate Carbon (after Vol.)[b]	% Decomposition[c]
		Before Vol.	After Vol.			
104	Res. (Fines)[d]	0.56	0.28	15.86	0.24	57.14
106	"	0.76	0.70	12.55	0.61	19.74
108	"	0.52	0.34	12.71	0.30	42.31
110	"	0.70	0.55	11.84	0.48	31.43
112	"	0.93	0.75	25.30	0.56	39.78
171	"	0.40	0.38	2.68	0.37	7.50
173	"	0.15	0.14	0.19	0.14	6.67
438	"	0.15	0.09	7.54	0.08	46.66
439	"	0.07	0.09	3.05	0.09	0.00
440	"	0.05	0.04	3.98	0.04	20.00
446	"	0.17	0.03	13.63	0.03	82.35
447	"	0.16	0.02	5.61	0.02	87.50
448	"	0.42	0.05	9.94	0.04	90.47
10	"	0.65	0.50	2.44	0.49	24.61
13	"	0.58	0.62	1.81	0.61	0.00
15	"	0.42	0.31	1.32	0.31	26.19
17	"	0.51	0.46	1.50	0.45	11.76
18	"	1.24	0.82	25.08	0.61	50.80
113	Res. (Organics)[e]	0.50	0.52	60.70	0.20	60.00
170	"	0.14	0.12	67.43	0.04	71.43
172	"	0.14	0.75	84.53	0.12	14.29
11	"	0.64	0.38	31.93	0.26	59.37
14	"	7.91	6.81	0.71	6.76	14.53
105	"	0.44	0.22	51.32	0.11	75.00
107	"	0.40	0.48	55.77	0.21	47.50
109	"	0.18	0.08*	63.93	0.03	≈83.33
111	"	0.50	0.24	49.23	0.12	76.00

TABLE I (continued)

CARBONATE DECOMPOSITION IN VOLATILE ANALYSIS
(2 hrs. @ 600 C in air)[a]

Sample No.	Type of Sample	% Carbonate Carbon		% Vol.	Corrected % Carbonate Carbon (after Vol.)[b]	% Decomposition[c]
		Before Vol.	After Vol.			
19	Fly Ash	0.94	0.06*	7.12	0.06	≈93.61
20	"	1.08	0.06*	6.18	0.06	≈94.44
21	"	1.16	0.08*	6.76	0.07	≈93.96
22	"	1.23	0.08*	6.32	0.07	≈94.30
24	"	0.96	0.10*	6.56	0.09	≈90.62
90	"	0.14	0.01*	1.84	<0.01	≈100.00
91	"	0.08*	0.01*	5.20	<0.01	≈100.00
92	"	0.15	0.04*	4.68	0.04	≈73.33
93	"	0.12*	0.04*	3.90	0.04	≈66.66
94	"	0.11*	0.02*	1.81	0.02	≈81.81
96	"	0.35	0.21	2.43	0.20	42.86
97	"	0.15	0.10	4.89	0.10	≈33.33
99	Raw Refuse	0.10	0.32	87.26	0.04	60.00
100	"	0.14	0.12	88.80	0.01	92.85
101	"	0.16	0.08	86.57	0.01	93.75
102	"	0.16	0.36	84.37	0.06	62.50
103	"	0.10	0.08	91.46	<0.01	100.00
165	"	0.03*	0.43	91.79	0.04	≈0.00
166	"	0.09	0.31	86.45	0.04	55.56
167	"	0.08	0.37	90.76	0.03	62.50
169	"	0.04*	0.44	94.89	0.02	≈50.00
434	"	0.25	0.20	90.02	0.02	92.00
435	"	0.42	0.29	95.90	0.01	97.61
436	"	0.65	0.27	94.41	0.02	96.92
437	"	0.50	0.10	93.77	<0.01	100.00
442	"	0.31	0.18	84.92	0.03	90.32
443	"	0.47	0.46	90.98	0.04	91.48

TABLE I (continued)

CARBONATE DECOMPOSITION IN VOLATILE ANALYSIS
(2 hrs. @ 600 C in air)[a]

Sample No.	Type of Sample	% Carbonate Carbon Before Vol.	% Carbonate Carbon After Vol.	% Vol.	Corrected % Carbonate Carbon (after Vol.)[b]	% Decomposition[c]
444	Raw Refuse	0.72	0.19	93.95	0.01	98.61
445	"	0.83	0.12	93.87	<0.01	100.00
5	"	0.09	0.08	86.72	0.01	88.88
7	"	0.72	0.35	87.61	0.04	94.44
9	"	0.31	0.27	85.18	0.04	87.09
256	Compost	1.14	0.51	43.03	0.29	74.56
257	"	1.09	0.51	43.02	0.29	73.39
258	"	2.82	3.99	41.66	2.33	17.38
288-D	"	2.74	3.89	41.61	2.27	17.15

*Values are below precision limits of method.

[a]All results are on a dry basis. Values have been converted from a wet to dry basis by the following formula:

$$\%\ \text{value (dry basis)} = \frac{(100)(\%\ \text{value})}{(100-\%\ \text{moisture})}\ \text{(wet basis)}$$

Except volatile data:

$$\%\ \text{volatile, dry} = (\text{total \% volatile, wet}, -\%\ \text{moisture}) + (100-\%\ \text{moisture})$$

[b]Results have been corrected to original sample basis by the following formula:

$$\text{corrected \% carbonate carbon (after Vol.)} = (\%\ \text{carbonate carbon found})(\%\ \text{ash in ash after vol.})$$

[c]Percent decomposition calculated using the following formula:

$$\%\ \text{decomposition} = \frac{(\%\ \text{carbonate carbon (before Vol.)} - \text{corrected \% carbonate carbon (after Vol.)})(100)}{\%\ \text{carbonate carbon (before Vol.)}}$$

[d]Residue "Fines" or "Noncombustibles" are materials remaining after most of the readily combustible substances have been removed by manual sorting. The sorting was performed at the incinerator cites and a 1/2-inch sieve was employed to asist in the separation.

[e]Residue "Organics" or "Combustibles" are mostly the readily combustible materials. Unlike "Fines" these materials are usually retained on a 1/2-inch sieve.

TABLE II

CARBONATE DECOMPOSITION IN VOLATILE ANALYSIS
(FROM CARBON-HYDROGEN METHOD)[a]

Sample No.	Type of Sample	% Carbonate Carbon		% Vol.	Corrected % Carbonate Carbon (After Vol.)	% Decomposition
		Before Vol.	After Vol.			
91	Fly Ash	0.08	0.02*	6.16	0.02	≈100.00
92	Fly Ash	0.15	0.02*	3.39	0.02	≈100.00
93	Fly Ash	0.12	0.02*	4.24	0.02	≈100.00
103	Raw Refuse	0.10	0.06	92.44	<0.01	100.00
165	Raw Refuse	0.03*	0.18	94.32	0.01	≈100.00
166	Raw Refuse	0.09	0.05	88.19	<0.01	100.00

* Values are below detection limit of method.

[a] All values are on a dry basis.

TABLE III

CARBONATE DECOMPOSITION IN VOLATILE ANALYSIS
(Muffled* 1 hr. @ 950 C in air)[a]

Sample No.	Type of Sample	% Carbonate Carbon Before Vol.	% Carbonate Carbon After Vol.	% Vol.	Corrected % Carbonate Carbon (After Vol.)	% Decomposition
104	Res. (Fines)	0.56	0.02	16.55	0.02	96.42
106	Res. (Fines)	0.76	0.04	13.22	0.03	96.05
173	Res. (Fines)	0.15	<0.01**	0.00	<0.01	≈100.00
105	Res. (Organic)	0.44	0.02**	53.90	<0.01	≈100.00
107	Res. (Organic)	0.40	0.03**	56.70	0.01	≈100.00
92	Fly Ash	0.15	0.02**	3.76	0.02	≈100.00
94	Fly Ash	0.11**	0.01**	0.14	<0.01	≈100.00
96	Fly Ash	0.35	0.01**	1.69	<0.01	≈100.00
101	Raw Refuse	0.16	0.04**	84.42	<0.01	≈100.00
102	Raw Refuse	0.16	0.02**	86.65	<0.01	≈100.00

* Lindberg Hevi-Duty, box type, furnace employed. However, product (or manufacturer) identification shown in this report does not imply endorsement by Environmental Protection Agency.

** Values are below detection limit of method.

[a] All results are on a dry basis.

TABLE IV

COMPARISON OF PERCENT VOLATILE DATA BY THE CARBON-HYDROGEN METHOD AND THE MUFFLE FURNACE METHOD (1 hr. @ 950 C in air)

Sample No.	Type of Sample	Percent Volatile[a]	
		Carbon-Hydrogen [b]	Muffle Furnace [c]
104	Res. (Fines)	16.24	16.56
106	Res. (Fines)	13.26	13.22
173	Res. (Fines)	0.31	0.00
105	Res. (Organics)	55.58	53.90
107	Res. (Organics)	57.38	56.70
92	Fly Ash	3.39	3.76
96	Fly Ash	1.90	1.69
102	Raw Refuse	88.16	86.65

[a] All results are on a dry basis.

[b] Values represent the average of two observations.

[c] Values represent the average of two to six observations.

TABLE V

VARIATION IN VOLATILE ANALYSIS (2 hrs. @ 600 C in air)
WITH AGE OF SAMPLE[a]

Sample No.	Type of Sample	First Analysis			Second Analysis[b]		
		Approx. Date	No. of Samples	Ave. Vol.	Approx. Age	No. of Samples	Ave. Vol.
438	Res. (Fines)	1/69	3	7.67	2 years	2	7.54
439	"	"	3	3.26	"	2	3.05
440	"	"	3	4.10	"	2	3.98
446	"	"	3	13.89	"	2	13.63
447	"	"	3	5.89	"	2	5.61
448	"	"	3	10.15	"	2	9.94
10*	"	4/69	3	2.11	1 yr. 8 months	2	2.44
13*	"	"	3	0.53	"	2	1.81
15*	"	"	3	0.79	"	2	1.32
17*	"	"	3	1.05	"	2	1.50
11*	Res. (Organics)	"	3	32.51	"	2	31.93
14**	"	"	2	2.16	"	2	0.71
18**	"	"	3	25.21	"	2	25.08
105*	"	2/70	3	51.96	10 months	4	51.32
107*	"	"	3	56.87	"	2	55.77
109	"	"	3	63.88	"	3	63.93
111*	"	"	3	49.14	"	2	49.23
113*	"	"	3	62.12	"	3	60.70
19**	Fly Ash	4/69	3	5.58	1 yr. 8 months	2	7.12
20**	"	"	3	4.01	"	2	6.18
21**	"	"	3	2.48	"	2	6.75

TABLE V (continued)

VARIATION IN VOLATILE ANALYSIS (2 hrs. @ 600 C in air)
WITH AGE OF SAMPLE[a]

Sample No.	Type of Sample	First Analysis			Second Analysis[b]		
		Approx. Date	No. of Samples	Ave. Vol.	Approx. Age	No. of Samples	Ave. Vol.
22**	Fly Ash	4/69	3	3.82	1 yr. 8 months	2	6.32
24**	"	"	3	3.22	"	2	6.56
90	"	2/70	3	1.81	10 months	2	1.84
91	"	"	3	5.04	"	2	5.20
92	"	"	3	4.52	"	2	4.68
93	"	"	3	3.83	"	2	3.90
94	"	"	3	1.77	"	2	1.81
96	"	"	3	2.75	"	2	2.43
97	"	"	3	4.59	"	2	4.89
434	Raw Refuse	1/69	3	89.66	2 years	7	90.02
435	"	"	3	95.72	"	6	95.90
436	"	"	3	94.47	"	5	94.41
437	"	"	3	93.71	"	4	93.77
442	"	"	3	84.54	"	3	84.92
443	"	"	3	90.20	"	6	90.98
444	"	"	3	94.66	"	6	93.95
445	"	"	3	93.84	"	6	93.87

TABLE V (continued)

VARIATION IN VOLATILE ANALYSIS (2 hrs. @ 600 C in air)
WITH AGE OF SAMPLE[a]

Sample No.	Type of Sample	First Analysis			Second Analysis[b]		
		Approx. Date	No. of Samples	Ave. Vol.	Approx. Age	No. of Samples	Ave. Vol.
5	Raw Refuse	4/69	3	86.60	1 yr. 8 months	4	86.72
7	"	"	3	86.96	"	5	87.61
8	"	"	3	90.81	"	6	90.81
9	"	"	3	85.50	"	5	85.18
99	"	2/70	3	87.60	10 months	6	87.26
100	"	"	3	89.06	"	6	88.80
101	"	"	3	86.25	"	5	86.57
102	"	"	3	87.13	"	6	84.37
103	"	"	3	91.66	"	6	91.46

* Residue or fly ash samples containing 0.4% to 0.9% carbonate carbon.

** Residue or fly ash samples containing 0.9% to 7.9% carbonate carbon.

[a] All results are on a dry basis.

[b] Samples stored dry, in screw-top glass or plastic jars, and without control of fluctuation in normal room temperature.

TABLE VI

COMPARISON OF TOTAL PERCENT OXYGEN VALUES USING THE THREE FORMULAS DESCRIBED HEREIN

Sample No.	Type of Sample	Total Percent Oxygen[a]		
		ASTM[b]	Volatiles @ 950 C[c]	Volatiles @ 600 C
171	Res. (Fines)	0.00	0.70	1.00
173	"	0.00	0.00	0.00
438	"	0.02	---[d]	0.40
448	"	0.00	---	0.17
10	"	0.00	---	2.42
13	"	0.06	---	2.86
15	"	0.00	---	1.33
17	"	0.00	---	2.10
106	"	2.99	---	6.22
108	"	2.22	3.40	3.93
110	"	3.15	---	5.74
112	"	5.87	---	9.06
18	Res. (Organics)	10.18	---	13.57
11	"	12.29	---	13.60
14	"	0.00	---	25.90
107	"	16.45	18.06	17.25
111	"	15.69	16.57	16.30
19	Fly Ash	2.22	---	3.59
20	"	2,08	---	3.64
21	"	1.99	---	3.69
22	"	2,45	---	4.25
24	"	3,12	---	4.63
93	"	0,39	0,63	0,60
96	"	0.53	---	1.63
97	"	0,70	---	1,17
7	Raw Refuse	36,14	---	36,75
9	"	34,84	---	34,88

TABLE VI (continued)

COMPARISON OF TOTAL PERCENT OXYGEN VALUES USING
THE THREE FORMULAS DESCRIBED HEREIN

Sample No.	Type of Sample	Total Percent Oxygen[a]		
		ASTM[b]	Volatiles @ 950 C[c]	Volatiles @ 600 C
166	Raw Refuse	34.93	36.40	34.93
436	"	40.13	---	40.52
437	"	39.90	---	40.05
442	"	31.30	---	31.42
443	"	39.47	---	39.70
444	"	39.84	---	40.55
445	"	38.45	---	39.26
256	Compost[e]	17.00	18.27	19.08
257	"	16.68	18.65	18.70
258	"	9.99	18.84	21.79
258-D	"	10.22	18.45	21.74

[a] All results are on a dry basis.

[b] Data obtained with the normal procedure of volatilizing solid wastes samples at 600 C was used in this formula. NOTE: Values do not include chlorine concentrations.

[c] Data obtained with volatilizing solid wastes samples as part of the carbon-hydrogen analyses was used in this formula.

[d] Since not all volatile @ 950 C data is available, total percent oxygen content could not be calculated for some samples.

[e] Oxygen values for compost samples are determined similar to the calculation of oxygen values for raw refuse samples.

LITERATURE CITED

American Public Works Association, in "Municipal Refuse Disposal", 3rd ed., pp. 393-5, Public Administration Service, Chicago, 1970.

American Society for Testing Materials, in "1969 Book of ASTM Standards; Including Tentatives," Pt. 19, p. 35, Philadelphia, 1969a.

American Society for Testing Materials, in "1969 Book of ASTM Standards; Including Tentatives," Pt. 19, pp. 19-20, Philadelphia, 1969b.

American Society for Testing Materials, in "1969 Book of ASTM Standards; Including Tentatives." Pt. 19, pp. 27-32, Philadelphia, 1969c.

American Society for Testing Materials, in "1969 Book of ASTM Standards; Including Tentatives," Pt. 19, pp. 25-26, Philadelphia, 1969d.

Association of Official Analytical Chemists, in "Official Methods of Analysis of the Association of Official Analytical Chemists," 11th ed., pp. 16-20, Washington, D.C., 1970a.

Association of Official Analytical Chemists, in "Official Methods of Analysis of the Association of Official Analytical Chemists," 11th ed., p. 602, Washington, D.C., 1970b.

Wilson, D. L., "Laboratory Procedure for the Gravimetric Determination of Carbon and Hydrogen in Solid Wastes," open-file report (RS-03-68-17). Solid Waste Management Office, Cincinnati, Ohio 1970.

Wilson, D. L., "Laboratory Procedure for the Gravimetric Determination of Carbonate Carbon in Solid Wastes," open-file report (RS-03-68-17). Solid Waste Management Office, Cincinnati, Ohio, 1971.

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